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VACUUM CARBOTHERMAL PROCESSING OF LOW-IRON BAUXITES

B. A. Goldin,[1] V. E. Grass,[1] and Yu. I. Ryabkov[1]

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The sequence of the phase formation processes in carbothermal processing of low-iron bauxites in a vacuum is investigated. A new technology for dry firing treatment of natural aluminosilicate material is offered. The possibility of production of a wide range of products is suggested.

The Republic of Komi has a leading place among the known bauxite deposits in Russia and CIS countries. The total reserves of the Vorykvinskoe group of deposits are sufficient for 40-year operation of a mine which is being designed here with expected annual production of 6 million tons of bauxites. Moreover, a substantial portion of the central bed of the Vezhayu-Vorykvinskoe deposit which is the largest in the Timan region consists of naturally decolorized low-iron bauxites, whose reserves are estimated at over 43 million tons.

The most common ore types here are boehmite and kaolinite-boehmite. The high quality of bauxites of the first type containing up to 75 – 78% Al_2O_3 with a silicon modulus equal to 50 and more, makes it possible to use these ores in production of high-temperature refractories, abrasives, and structural ceramics. At the same time, a substantial part of the kaolin-boehmite bauxites are distinguished by a relatively high content of SiO_2 (18 – 20%) and, accordingly, are regarded as low-quality ores [1]. Therefore, to be used in ceramic production, these bauxites, as well as the ores occupying an intermediate position between low-modulus kaolinite-boehmite and high-quality boehmite ores, require concentration processing to increase their silicon modulus.

[1] Institute of Chemistry, Komi Research Center, Ural Branch, the Russian Academy of Sciences, Russia.

The traditional methods for bauxite processing (Bayer method, sintering method, hydrochemical methods, various combinations of methods) [2, 3] make it possible to obtain

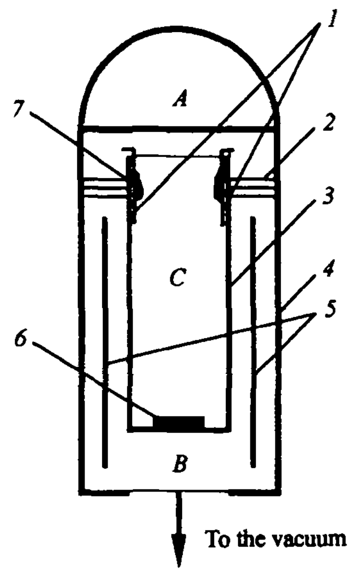

Fig. 1. Scheme of experimental plant. *1*) Metal plates for recovery of condensation products, *2*) heat-insulating screen; *3*) corundum tube; *4*) case; *5*) heaters, *6*) briquetted bauxite-carbon mixture; *7*) condensate. *A*) cold zone, *B*) hot zone, *C*) reaction chamber.

TABLE 1

Kaolinite-boehmite bauxites	Weight content, %*										
	Al_2O_3	SiO_2	TiO_2	$Fe_2O_3 + FeO$	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	Calcination loss
Before calcination	64.79	13.16	3.98	2.48	0.01	0.21	0.10	0.13	0.36	0.09	13.99
After calcination	75.34	15.30	4.63	2.88	0.01	0.25	0.12	0.15	0.42	0.13	0.54

* Relative precision of chemical analysis of 2%.

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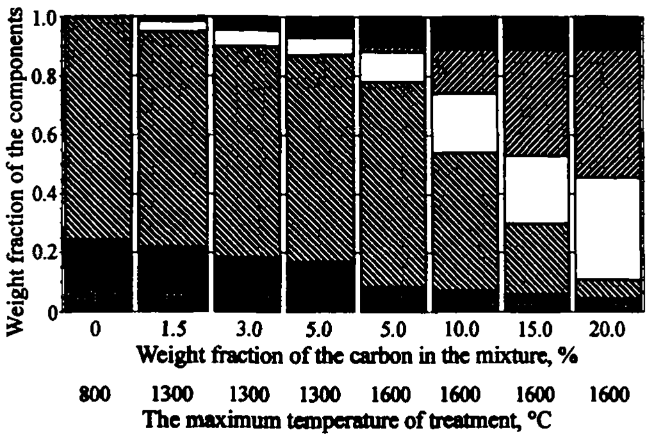

Fig. 2. Variation in the weight fraction of the main components as the result of high-temperature vacuum treatment of bauxite-carbon mixtures. ▩) Iron-containing products, ■) silicon-containing products; □) removed gaseous products, ■) silicon-containing products (condensate); ■) titanium-containing products; ▧) aluminum-containing products; ▨) aluminum-containing products (condensate).

alumina with a minimum amount of impurities but have a number of significant drawbacks: numerous technological stages involving aggressive reactants, the need for utilization of substantial quantities of environmentally unsafe wastes, etc. It should be also noted that in many cases deep refinement of bauxites does not make sense. In particular, SiO_2, TiO_2, and other additives often are obligatory components in production of ceramic articles based on Al_2O_3 [4].

Alongside the above alkaline methods, carbothermal treatment of oxide materials is well known and widely used in powder metallurgy for carbide production. In the course of such treatment of bauxites, perceptible quantities of the resulting silicon and aluminum suboxides can pass into the gas phase. Since these elements have different chemical affinity with respect to oxygen, the thermodynamic strength of their oxide compounds is not equal, and therefore different conditions are required for reducing reactions to proceed [5]. This can be used as the basis for a new method for separation of the components in natural aluminosilicate systems. In this case, vacuum conditions are optimum for carbothermal treatment: on one side, the resulting gaseous products are efficiently removed, and on the other side, the optimum protective medium for the suboxide and carbide phases is provided [4, 6].

As a result of the experiments performed by the authors, a promising scheme for vacuum-carbothermal reducing processing of low-iron bauxites was proposed.

The preparation of raw material for high-temperature vacuum treatment included its calcination in air at a temperature about 800°C up to removal of chemically bound water (the chemical composition of the low-iron kaolinite-boehmite bauxites from the Vezhayu-Varykvinskoe deposit before and after calcination is shown in Table 1), mixing with carbon (milled activated charcoal of BAU-A grade) and briquetting of the mixtures under a load of 40 kN.

The compacted mixtures were heat-treated in vacuum (the residual pressure in the working chamber was $10^{-3} - 10^{-2}$ Pa, the temperature was 1300 – 1600°C) on a experimental plant designed on the basis of a furnace of the SShVE-1.2.5/25-I2 type (Fig. 1). At the same time, the dependence of the pressure variation in the reaction chamber on the temperature-time factor was recorded.

The heat treatment of the mixtures in a vacuum was accompanied by intense gas emission. The pressure increase inside the reaction chamber mainly proceeded due to the emergence of gaseous suboxides of carbon, silicon, and alu-

TABLE 2

Carbon content in mixture, wt.%	Maximum temperature of treatment, °C	Phase composition of products	
		not converted to the gas phase	condensed in the cold zone
0	800 (calcination)	Boehmite γ-AlO[OH], kaolinite $Al_2[OH]_4(Si_2O_5)$	–
1.5	1300	Corundum α-Al_2O_3, mullite $3Al_2O_3 \cdot 2SiO_2$, Ti_2O_3, spinel phase (Al, Ti, Fe, ...)O · Al_2O_3	X-ray amorphous silicon compounds
3.0	1300	Corundum α-Al_2O_3, mullite $3Al_2O_3 \cdot 2SiO_2$, Ti_2O_3, spinel phase (Al, Ti, Fe, ...)O · Al_2O_3, FeO	The same
5.0	1300	Corundum α-Al_2O_3, Ti_2O_3, spinel phase (Al, Ti, Fe, ...)O · Al_2O_3, FeO	X-ray amorphous silicon and aluminum compounds
5.0	1600	Corundum α-Al_2O_3, Ti_2O_3, FeO	Silicon- and aluminum-containing phases
10.0	1600	Corundum α-Al_2O_3, Ti_2O_3, TiO_xC_y, FeO	The same
15.0	1600	Corundum α-Al_2O_3, Ti_2O_3, TiO_xC_y, FeO	Phases containing silicon and aluminum with traces of aluminum and silicon carbide phases
20.0	1600	Corundum α-Al_2O_3, TiC, FeO	β-SiC, Al_4O_4C, Al, Al_4C_3, α-Al_2O_3

minum in the course of synthesis, according to the following reactions:

$$SiO_{2sol} + C_{sol} = SiO_{gas} + CO_{gas};$$

$$Al_2O_{3sol} + 2C_{sol} = Al_2O_{gas} + 2CO_{gas}.$$

As the reducing reactions were completed, the emission of gas ended, and the initial pressure values were restored.

The weight losses and the variations in the chemical and phase composition of the mixtures and the condensate which was formed due to the following reactions:

$$SiO_{gas} = SiO_{sol};$$

$$3Al_2O_{gas} = 4Al_{sol} + Al_2O_{3sol};$$

$$2Al_{sol} + CO_{gas} = Al_2OC_{sol};$$

$$4Al_2OC_{sol} = Al_4O_4C_{sol} + Al_4C_{3sol};$$

$$Al_2O_{gas} + 2CO_{gas} = Al_2O_{3sol} + 2C_{sol};$$

$$2Al_2O_{gas} + 2CO_{gas} = Al_4O_4C_{sol} + C_{sol},$$

on the parts located in the cold zone of the reaction chamber are shown in Table 2 and in Fig. 2.

The experimental results interpreted in conformity with the published data on the interaction between metal oxides and carbon [4, 5, 7] enable us to infer the possibility of using vacuum carbothermal processing for the separation of Si- and Al-bearing bauxite components. At the same time, the quantity of reduced (and removed from the initial material) SiO_2 is proportional to the carbon content in the mixture, which makes it possible to control the silicon modulus of the intermediate bauxite product and, consequently, on its basis to obtain end products (e.g., corundum ceramics) with preassigned physicochemical properties.

It ought to be noted that vacuum carbothermal treatment of low-iron bauxites produced oxide, carbide, and oxycarbide compounds of aluminum, silicon, and titanium, which were analyzed using the x-ray diffraction and spectral methods (Table 2). These compounds can be isolated or synthesized in the required ratios and, therefore, are of independent significance as objects of fundamental and applied research and as commodities that can be components of ceramic, refractory, abrasive and other technical materials.

The upgrading of the system of recovery (condensation) of gaseous reaction products will make it possible to convert the process into virtually wasteless technology (see Fig. 3).

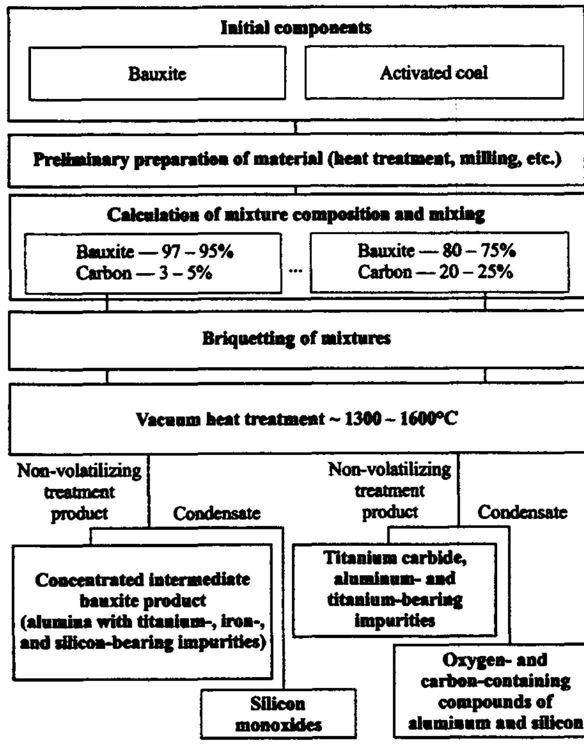

Fig. 3. Scheme of vacuum carbothermal treatment of low-iron bauxites.

Thus, the vacuum carbothermal process opens wide prospects for the development of efficient technologies for processing of aluminosilicate, and in particliuar, raw bauxites, which allows for production of a wide range of commercial products.

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